

FORM-PTO-1390
(Rev. 9-2001)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

000510-011

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5)

10/069873INTERNATIONAL APPLICATION NO.
PCT/SE00/01670INTERNATIONAL FILING DATE
31 August 2000PRIORITY DATE CLAIMED
01 September 1999TITLE OF INVENTION
FIBER BONDING

APPLICANT(S) FOR DO/EO/US

Lars Fingal and Anders Strålin


Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). **(unexecuted)**
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information: International Preliminary Examination Report

**21839**

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.51) 10/069873		INTERNATIONAL APPLICATION NO. PCT/SE00/01670		ATTORNEY'S DOCKET NUMBER 000510-011	
21. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$1,040.00 (960) International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$890.00 (970) International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$740.00 (958) International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$710.00 (956) International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 (962)					
ENTER APPROPRIATE BASIC FEE AMOUNT =					
Surcharge of \$130.00 (154) for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492(e)). 20 <input type="checkbox"/> 30 <input type="checkbox"/>				\$	
Claims	Number Filed	Number Extra	Rate		
Total Claims	22 -20 =	2	X\$18.00 (966)	\$ 36.00	
Independent Claims	1 -3 =	0	X\$84.00 (964)	\$	
Multiple dependent claim(s) (if applicable)			+ \$280.00 (968)	\$	
TOTAL OF ABOVE CALCULATIONS =				\$ 926.00	
Reduction for 1/2 for filing by small entity, if applicable (see below). +				\$	-
SUBTOTAL =				\$ 926.00	
Processing fee of \$130.00 (156) for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)). 20 <input type="checkbox"/> 30 <input type="checkbox"/> +				\$	
TOTAL NATIONAL FEE =				\$ 926.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 (581) per property +				\$	
TOTAL FEES ENCLOSED =				\$ 926.00	
				Amount to be refunded:	\$
				charged:	\$
a. <input type="checkbox"/> Small entity status is hereby claimed. b. <input checked="" type="checkbox"/> A check in the amount of \$ <u>926.00</u> to cover the above fees is enclosed. c. <input type="checkbox"/> Please charge my Deposit Account No. <u>02-4800</u> in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed. d. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>02-4800</u> . A duplicate copy of this sheet is enclosed. NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO: Ronald L. Grudziecki BURNS, DOANE, SWECKER & MATHIS, L.L.P. P.O. Box 1404 Alexandria, Virginia 22313-1404 (703) 836-6620					
			 SIGNATURE William C. Rowland NAME 30,888 REGISTRATION NUMBER		
			March 1, 2002 DATE		

Patent
Attorney's Docket No. 000510-011

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)
Lars FINGAL et al) Group Art Unit: Unassigned
Application No.: Unassigned) Examiner: Unassigned
(Corresponds to PCT/SE00/01670)
Filing Date: March 1, 2002)
For: FIBER BONDING)

PRELIMINARY AMENDMENT

BOX PCT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to examining the above-identified application, please amend the application as follows:

IN THE CLAIMS:

Please amend claims 1-17 as follows:

1. (Amended) A method of hydroentangling polymer fibers to manufacture a nonwoven fabric, comprising imparting to the polymer fibers, at the moment of hydroentangling, a temperature equal to or exceeding the glass transition temperature (T_g) of the polymer fiber and being less than the melting point of the polymer fiber.
2. (Amended) Method according to claim 1, wherein the polymer fiber has an initial modulus ≥ 50 cN/tex, at room temperature.

3. (Amended) Method according to claim 1, wherein the polymer fiber has an initial modulus ≥ 100 cN/tex, at room temperature.

4. (Amended) Method according to claim 3, wherein the polymer fiber has an initial modulus of 100-2000 cN/tex, at room temperature.

5. (Amended) Method according to claim 1, wherein the temperature is achieved with the aid of hot or superheated water.

6. (Amended) Method according to claim 1, wherein the temperature is achieved with the aid of IR-heat.

7. (Amended) Method according to claim 1, wherein the temperature is achieved with the aid of microwaves.

8. (Amended) Method according to claim 1, wherein the polymer fiber has a glass transition temperature (T_g) of $\geq 20^{\circ}\text{C}$.

9. (Amended) Method according to claim 1, wherein the polymer fiber has a glass transition temperature (T_g) of ≥ 20 - 100°C .

10. (Amended) Method according to claim 1, wherein the polymer included in the polymer fibers comprises polyester, polylactic acid, polyamide or polypropylene, or copolymers or mixtures thereof.

11. (Amended) A hydroentangled nonwoven fabric obtainable by the method of claim 1, the fabric comprising polymer fibers, wherein the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of 20-100°C and an initial modulus of 200-750 cN/tex at room temperature.

12. (Amended) Nonwoven fabric according to Claim 11, wherein the polymer fibers in the nonwoven fabric have an initial modulus of 250-600 cN/tex at room temperature.

13. (Amended) Nonwoven fabric according to claim 11, wherein the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of 50-70°C.

14. (Amended) Nonwoven fabric according to claim 11, wherein the nonwoven fabric has a bulk specific volume of $\geq 8 \text{ cm}^3/\text{g}$.

15. (Amended) Nonwoven fabric according to claim 14, wherein the nonwoven fabric has a bulk specific volume of 8-15 cm^3/g .

16. (Amended) Nonwoven fabric according to claim 11, wherein the polymer included in the polymer fibers comprises polyester, polylactic acid, poly-amide or polypropylene, or copolymers or mixtures thereof.

17. (Amended) Nonwoven fabric according to claim 11, wherein the polymer included in the polymer fibers comprises polyester, polylactic acid, poly-amide or polypropylene, or copolymers or mixtures thereof.

Please add the following new claims:

18. (New) Method according to claim 3, wherein the polymer fiber has an initial modulus of 500-1500 cN/tex at room temperature.

19. (New) Method according to claim 3, wherein the polymer fiber has an initial modulus of 200-750 cN/tex at room temperature.

20. (New) Method according to claim 3, wherein the polymer fiber has an initial modulus of 250-600 cN/tex at room temperature.

21. (New) Method according to claim 1, wherein the polymer fiber has a glass transition temperature (T_g) of 50-70°C.

22. (New) Nonwoven fabric according to claim 14, wherein the nonwoven fabric has a bulk specific volume of 10-15 cm³/g.

000510-011

REMARKS

The foregoing amendments have been made to remove multiple claim dependencies, and to otherwise put the claims in the preferred U.S. format.

In the event that there are any questions concerning this Amendment, or the application in general, please contact the undersigned so that prosecution of the application may be expedited.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: William C. Rowland

William C. Rowland
Registration No. 30,888

P.O. Box 1404
Alexandria, Virginia 22313-1404
(703) 836-6620

Date: March 1, 2002

Attachment to Preliminary Amendment dated March 1, 2002

Marked-Up Claims

1. (Amended) [Method] A method of hydroentangling polymer fibers to manufacture a nonwoven fabric, [characterized in that] comprising imparting to the polymer [fiber] fibers, at the moment of hydroentangling, [is imparted] a temperature equal to or exceeding the glass transition temperature (Tg) of the polymer fiber and being less than the melting point of the polymer fiber.
2. (Amended) Method according to claim 1, [characterized in that] wherein the polymer fiber has an initial modulus ≥ 50 cN/tex, at room temperature.
3. (Amended) Method according to claim 1, [characterized in that] wherein the polymer fiber has an initial modulus ≥ 100 cN/tex, at room temperature.
4. (Amended) Method according to claim 3, [characterized in that] wherein the polymer fiber has an initial modulus of 100-2000 cN/tex[, especially 500-1500 cN/tex, more particularly 200-750 cN/tex, and even more particularly 250-600 cN/tex], at room temperature.
5. (Amended) Method according to [any of claims 1-4, characterized in that] claim 1, wherein the temperature is achieved with the aid of hot or superheated water.

Application No. Unassigned
Attorney's Docket No. 000510-011
Mark-up of Claims - Page 1

Attachment to Preliminary Amendment dated March 1, 2002

Marked-Up Claims

1. (Amended) [Method] A method of hydroentangling polymer fibers to manufacture a nonwoven fabric, [characterized in that] comprising imparting to the polymer [fiber] fibers, at the moment of hydroentangling, [is imparted] a temperature equal to or exceeding the glass transition temperature (T_g) of the polymer fiber and being less than the melting point of the polymer fiber.
2. (Amended) Method according to claim 1, [characterized in that] wherein the polymer fiber has an initial modulus ≥ 50 cN/tex, at room temperature.
3. (Amended) Method according to claim 1, [characterized in that] wherein the polymer fiber has an initial modulus ≥ 100 cN/tex, at room temperature.
4. (Amended) Method according to claim 3, [characterized in that] wherein the polymer fiber has an initial modulus of 100-2000 cN/tex[, especially 500-1500 cN/tex, more particularly 200-750 cN/tex, and even more particularly 250-600 cN/tex], at room temperature.
5. (Amended) Method according to [any of claims 1-4, characterized in that] claim 1, wherein the temperature is achieved with the aid of hot or superheated water.

Attachment to Preliminary Amendment dated March 1, 2002

Marked-Up Claims

6. (Amended) Method according to [any of claims 1-4, characterized in that] claim 1, wherein the temperature is achieved with the aid of IR-heat.
7. (Amended) Method according to [any of claims 1-4, characterized in that] claim 1, wherein the temperature is achieved with the aid of microwaves.
8. (Amended) Method according to [any of claims 1-7, characterized in that] claim 1, wherein the polymer fiber has a glass transition temperature (T_g) of $\geq 20^{\circ}\text{C}$.
9. (Amended) Method according to [any of claims 1-8, characterized in that] claim 1, wherein the polymer fiber has a glass transition temperature (T_g) of $\geq 20\text{-}100^{\circ}\text{C}$ [, especially $50\text{-}70^{\circ}\text{C}$].
10. (Amended) Method according to [any of claims 1-9, characterized in that] claim 1, wherein the polymer included in the polymer fibers comprises polyester, polylactic acid, polyamide or polypropylene, or copolymers or mixtures thereof.
11. (Amended) [Hydroentangled] A hydroentangled nonwoven fabric obtainable by the method of [any of claims 1-10] claim 1, the fabric comprising polymer fibers, [characterized in that] wherein the polymer fibers in the nonwoven fabric have a glass

Attachment to Preliminary Amendment dated March 1, 2002

Marked-Up Claims

transition temperature (Tg) of 20-100°C and an initial modulus of 200-750 cN/tex at room temperature.

12. (Amended) Nonwoven fabric according to Claim 11, [characterized in that] wherein the polymer fibers in the nonwoven fabric have an initial modulus of 250-600 cN/tex at room temperature.

13. (Amended) Nonwoven fabric according to [any of claims 11-12, characterized in that] claim 11, wherein the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of 50-70°C.

14. (Amended) Nonwoven fabric according to [any of claims 11-13, characterized in that] claim 11, wherein the nonwoven fabric has a bulk specific volume of $\geq 8 \text{ cm}^3/\text{g}$.

15. (Amended) Nonwoven fabric according to claim 14, [characterized in that] wherein the nonwoven fabric has a bulk specific volume of $8\text{-}15 \text{ cm}^3/\text{g}$ [, especially $10\text{-}15 \text{ cm}^3/\text{g}$].

Attachment to Preliminary Amendment dated March 1, 2002

Marked-Up Claims

16. (Amended) Nonwoven fabric according to [any of claims 11-15, characterized in that] claim 11, wherein the polymer included in the polymer fibers comprises polyester, polylactic acid, poly-amide or polypropylene, or copolymers or mixtures thereof.

17. (Amended) Nonwoven fabric according to [one of claims 10-15, characterized in that] claim 11, wherein the polymer included in the polymer fibers comprises polyester, polylactic acid, poly-amide or polypropylene, or copolymers or mixtures thereof.

FIBER BONDING5 Technical field

The invention relates to a method for bonding polymer fibers into a nonwoven fabric and a nonwoven fabric manufactured with the aid thereof.

10 According to the invention the polymer fibers are subjected to hydroentangling, the fibers at the moment of hydroentangling being imparted a temperature exceeding the glass transition temperature of the polymer fiber and being less than the melting point of the polymer fiber.

15 Background of the invention

20 The manufacture of cloth and fabrics is very old art. In addition to the wellknown methods of weaving, knitting and crocheting, there are now a number of methods for the manufacture of nonwoven materials. Such materials can be made of both synthetic fibers and natural fibers. A number of methods utilize heat to melt the fibers together, so-called thermobonding.

25 There are also other bonding methods, such as stitch-bonding and hydroentangling or spun-lacing. Hydroentangling or spun-lacing is a technology which was introduced in the nineteen seventies. The method involves the forming of a fiber web, either dry-laid or wet-laid, whereafter the fibers are entangled together with the aid of very fine jets of water under high pressure. Several rows of water jets are directed towards the fiber web, which is carried by a moving wire or drum. Finally, the entangled fiber web is dried.

30 In hydroentangled materials many different types and mixtures of fibers are used, such as for example synthetic staple fibers, synthetic continuous fibers, staple fibers

of regenerated cellulose and pulp fibers. Examples of commercially available fibers of regenerated cellulose are rayon, viscose fibers and lyocell.

Hydroentangling is a method of bonding fibers without using bonding agents or bonding fibers. Hydroentangled material or spun-lace material of high quality can be made at reasonable cost, and has a high absorption capacity, good mechanical properties and a high textile comfort. They were used as wiping materials for house-hold or industrial use, as disposable materials in medical contexts and for hygiene etc.

In order to form a composite web by hydroentangling, the fibers to be entangled must have the right properties for this purpose.

One of several critical factors is the flexural rigidity of the fiber $= E \times I$, where E is the initial modulus of the fiber or the modulus of elasticity, and I is the moment of inertia. The moment of inertia is a sectional quantity ($I = \pi d^4/64$ for a circular cross section) which is dependent on the diameter of the fiber. The initial modulus is thus a material parameter, which is temperature-dependent.

A stiff fiber is more difficult to entangle and requires more specific energy (kWh/ton) to be bonded than a soft fiber, and this in turn limits the range of fibers which are technically and commercially of interest for use in this technology.

Fibers made of thermoplastic polymers, such as e.g. polypropylene, polyester, polyamide commonly occur in hydroentangling.

The properties of thermoplastic and other synthetic fibers are dependent on the properties of the component of polymer or polymers and the type of process used. Often the properties of the polymer cannot be fully utilized. Rather, a compromise must be made due to the technical constraints of the process.

It is typically very difficult to manufacture a fiber which is both very strong and at the same time has a low initial modulus. The strength of the fiber is largely determined by the orientation, imodulus follow the same tendency, i.e. a high fiber strength is accompanied by a high initial modulus.

5

CA 841 938 describes the manufacture of a nonwoven fabric by hydroentangling, whereby water under high pressure is pressed through a perforated carrier against a sheet of a fiber suspension, to give rise to entangling of the fibers.

WO 95/06769 describes a method and an apparatus for achieving a melt bond and possibly entangling of fibers in a fiber fabric, e.g. a nonwoven fabric. In this case, a steam jet or a jet of superheated steam is used to both melt and entangle the fibers. If one uses jets such as are normally used in hydroentangling, they must be sufficiently hot to melt a meltable component incorporated in the fiber web. WO95/06769 thus describes a process whereby a certain amount of melting of the meltable component is always achieved. This meltable component can either be the fibers themselves or an added meltable component in the form of a powder or granulate. No process for pure hydroentangling is described.

20 US 3 322 584 describes a melt-bonding process for bonding together two plastic webs. The method described can also be used to bond together two layers of plastic fibers, but this also refers to a melt bond, the temperature used being sufficiently high to melt the fibers.

25 US 5 069 735 describes a method of edge-melting adjacent sheets or fabrics to solve the problem that these sheets normally flock and are unsuitable for use in operations for example.

US 3 192 560 describes a method for controlled melt-bonding of fiber threads with a suitable medium, e.g. steam or superheated steam, the temperature being kept about or slightly under the melting point of the fiber threads.

5 One problem with hydroentangling is that the fiber components used must have a flexural rigidity such that the fibers can be entangled using reasonable energy levels. This means a limitation as regards the types of fibers used and means that thin fibers or fibers with a low initial modulus must be used, even if the fibers per se are not optimal for the formation of the fibrous web or for the functional properties of the finished material.

One purpose of the present invention is to achieve a process for manufacturing a nonwoven fabric by hydroentangling, where the flexural rigidity of the fiber components used does not limit the degree of entangling as much as previously.

An additional purpose of the present invention is to achieve a process making it possible to manufacture nonwoven fabrics having coarser fibers, consuming less energy or being stronger than what is possible with today's methods.

20 Another additional purpose of the present invention is to provide a nonwoven fabric with special properties, such as good mechanical properties, high bulk, etc.

Summary of the invention

25 We have now found that by raising the temperature at the very moment of entangling it is possible to reduce the flexural rigidity of the fibers and achieve a higher degree of entangling.

30 We have also found that it is only at the moment of entanglement that a too high initial modulus is of disadvantage. By reducing the initial modulus only during the entangling itself, and thereafter allowing the initial modulus to return to the original

level, a method and a material are provided having great advantages over those previously known.

5 There is less need to compromise in the fiber manufacturing process between the properties strength and rigidity. The strength of the fiber can instead be fully optimized.

At the same time it is possible to select fibers for the fabric to be bonded, based on other criteria than the limitations placed by the entangling process. In many cases it is an advantage to have stiff fibers in the finished fabric, depending on to what use the material is to be put.

Fibers already well adapted to hydroentangling can be further optimized, providing improved material properties and/or lower energy consumption in the process.

10
15
20 According to the invention, a process is achieved for hydroentangling of polymer fibers for producing a nonwoven fabric. The polymer fiber is imparted at the moment of hydroentangling, a temperature which is equal to or exceeds the glass transition temperature for the polymer fiber and is below the melting point for the polymer fiber.

25 According to the invention, there is further achieved a hydroentangled fabric comprising polymer fibers, the polymer fibers in the fabric having an initial modulus \geq 50 cN/tex .

Furthermore, according to the invention a hydroentangled polymer fiber fabric is obtained with a bulk specific volume of $\geq 8 \text{ cm}^3/\text{g}$.

30 Additional embodiments are disclosed in the accompanying subclaims.

Detailed description of the invention

According to the invention the polymer fiber is heated so that at the moment of entanglement it reaches a temperature above the glass transition temperature (T_g) of the polymer fiber. At this temperature the mobility in the molecules increases to such a degree that the stiffness is dramatically affected and a reduction in the modulus of elasticity or initial modulus by up to several powers of 10 can be obtained.

The mechanical properties of synthetic polymers are changed dramatically at the glass transition temperature of the polymer. By momentarily heating the desired fiber to the glass transition temperature or slightly thereabove at hydroentangling, the flexural rigidity and the degree of entangling in the fiber fabric are increased.

Many different types and mixtures of polymer fibers can be used. Of particular preference according to the invention is a nonwoven fabric which completely or partially comprises synthetic polymer fibers, or mixtures or copolymers of such polymer fibers. Based primarily on the purpose of the nonwoven fabric, the type of fiber and the percentage of natural fiber are selected. The greater the percentage of synthetic polymers included in the nonwoven fabric, the greater are the possibilities.

Examples of fibers which can be used in the material according to the present invention are synthetic staple fibers, synthetic continuous fibers, staple fibers of regenerated cellulose, natural fibers such as plant fibers, pulp fibers or mixtures thereof. Examples of commercially available fibers of regenerated cellulose are rayon, viscose and lyocell. Examples of synthetic fibers are fibers of polyester, polylactic acid, polyamide, polypropylene, polybutylene terephthalat (PBT), polyethylene (PE), polyethylene terephthalat (PET) and copolymers thereof, such as polyesteramides. Bicomponent fibers, i.e. fibers having a core of a first polymer, e.g. PET, and a casing of a second polymer, e.g. PE, may be used. The synthetic polymer fibers can comprise both polymer fibers made of natural fibers and polymer fibers made of

synthetic fibers. Even continuous filaments, such as melt-blown and spun-bond fibers can be used as well as profiled so-called capillary fibers. These profiled fibers are often very stiff and normally difficult to handle, but can be entangled with the aid of the present invention. Even mixtures of these different fibers can be used. A typical mixture is 40-50% long, synthetic fibers and the rest pulp, but all mixtures are applicable. The pulp

fibers can be of chemical, mechanical, thermomechanical, chemomechanical or chemothermomechanical pulp (CTMP). Mixing in of mechanical, thermomechanical, chemomechanical or chemothermomechanical pulp fibers provides a material with higher bulk and improved absorption and softness, which is described in SE 9500585-6.

According to the invention, primarily thermoplastic, synthetic polymers and in particular semi-crystalline polymers can be used. Amorphous polymers can also be used.

The heating of the polymer fiber at the moment of hydroentangling can be done in many different ways. One method of achieving a momentary temperature rise in the process is to heat the entangling water to such a temperature that the fiber, at the moment of hydroentangling reaches a temperature above T_g . Suitably this process is used when the T_g of the polymer lies below 100°C . This process can also be used at T_g -temperatures above 100°C , but will then require special equipment to produce superheated steam.

A further method of heating the fiber can be by IR-heating, e.g. by IR-radiation of the fiber web, or alternatively of the entangling water.

Other radiation heating can be used or microwave heating. A further possibility is to use metal wires, e.g. of copper, which is heated up with the aid of hot air, hot water or another medium or a combination thereof.

The hydroentangling can be done starting from either a dry-laid or a wet-laid fiber web. In dry-forming, the dry fibers are air-laid on a wire, whereafter the fiber web is subjected to hydroentangling. In wet-laying, a wet- or foam-formed web is made by the fibers being dispersed in liquid or in a foam liquid containing a foam-forming tenside and water. An example of a suitable foam-forming process of this type is described in SE 9402470-0. The fiber dispersion can be dewatered on a wire and thereafter be hydroentangled. The hydroentangling can be effected with conventional equipment.

Hydroentangling of a wet- or foam-formed fiber web can be done either in-line, i.e. directly when the fiber web has been dewatered on the wire, or on a wet-formed sheet which has been dried and rolled up after forming. Several such sheets can be laminated together by hydroentangling. It is also possible to combine dry-forming with wet- or foam-forming in such a way that an air-laid web of synthetic fibers, for example, is entangled together with a wet- or foam-formed paper sheet of pulp fibers.

After the hydroentangling, the material is pressed and dried and rolled up. The finished material can then be converted in a known manner to a suitable format and be packed.

According to one embodiment of the present process, a fiber dispersion is formed of the desired polymer fiber(s). The fiber dispersion is formed on a rotating carrier, e.g. a wire, and when the dispersion has been formed it is subjected to hydro-entangling by water jets striking against the layer of the fiber dispersion and in this manner entangling the fibers. At the moment of hydroentangling at least, the polymer fiber is imparted a temperature exceeding T_g for the polymer fiber, but at the same time being less than its melting point. This is done by heating the water used to produce the hydroentangling, at least during the hydroentangling itself, to a temperature

above T_g for the polymer fiber by one of the above ways. Typical energy levels used are 300 – 600 kWh/ton with a water pressure of 80 – 120 bar.

Preferably, the present invention is used for polymer fibers with a $T_g \geq 20^\circ\text{C}$,
5 especially 20 - 100°C , suitably 50 - 100°C and more particularly 50 - 70°C . T_g is preferably below 150°C . A particularly preferred polymer fiber is polylactic acid (PLA), which has a T_g of 50 - 70°C .

The reported glass transition temperature for a polymer can vary widely, on one hand due to the fact that glass transition occurs over a temperature interval and not at a certain temperature, and on the other hand depending on what method is used to determine the glass transition temperature.

One method which is usable in the present invention to determine the glass transition temperature is DSC (Differential Scanning Calorimetry), which measures the change in enthalpy as a function of temperature. At the glass transition temperature, the enthalpy-temperature curve makes a jump, and the value at this jump gives the glass transition temperature.

20 Another method which is considered more sensitive is DMA (Dynamic Mechanical Analysis). In this method the storage modulus, the loss modulus and $\tan\delta$ are measured at a frequency (normally 1 Hz) as a function of temperature. At the glass transition temperature, the storage module for an amorphous polymer changes by several orders of ten, while the loss module and $\tan\delta$ go through maximums. With
25 this method it is also possible to get an idea of how much the modulus changes at the glass transition temperature. The glass transition temperatures of most polymers are given in handbooks well-known to the person skilled in the art. For the purposes of the invention, T_g can be taken from "Polymer Handbook" by J. Brandrup and E.H. Immergut, publisher "Interscience Publishers". T_g can also be derived with the
30 aid of one of the methods DSC or DMA.

The method according to the invention is particularly suited to fibers with high flexural rigidity. High flexural rigidity can be achieved either by a high initial modulus value or by high fiber thickness. This means that polymer fibers especially suitable are either fibers with a high initial modulus or very thick polymer fibers, for example a thin fiber with a high initial modulus or a thick fiber with a less pronounced or low initial modulus. Alternatively, fibers which are both thick and have a high initial modulus can be used. The initial modulus for a polymer fiber is expressed in cN/tex.

Measurement of the initial modulus value for a fiber can for example be done by measuring the initial slope of a stress-strain diagram from a tensile test performed according to SS-EN ISO 5079. One example of the equipment, which can be used to measure the initial modulus according to the present invention, is a Lenzing Vibrodyn. With the aid of DMA it is also possible to get an idea of how much the modulus changes at the glass transition temperature. The initial modulus value for the polymer fiber is, in accordance with the present invention, the initial modulus value for the fiber at room temperature (see SS-EN ISO 5079).

Fibers of all thicknesses can be used, i.e. both microfibers, fibers of normal thickness about 1-2 dtex, and thick fibers about 6-7 dtex. According to a special embodiment, very thick fibers can be entangled to form a fiber fabric with high bulk.

According to the invention, new materials are also achieved, i.e. new nonwoven fabrics produced by hydroentangling.

Suitably, the polymer fiber has an initial modulus value ≥ 20 cN/tex, especially ≥ 50 cN/tex and most preferably ≥ 100 cN/tex. It is also possible to achieve nonwoven fabrics of polymers with very high initial modulus values, such as 100-2000 cN/tex, especially 500-1500 cN/tex, more particularly 200-750 cN/tex, and even more particularly 250-600 cN/tex.

According to one embodiment, the process according to the invention can be used to produce very strong nonwoven fabrics of fibers with very high initial modulus values, e.g. aromatic polyamides and aromatic polyesters.

5

Of special interest is the fact that according to the invention it is possible to manufacture nonwoven fabrics having a high bulk. With the aid of the present method, nonwoven fabrics having very thick fibers, e.g. 6-7 dtex, and which provide a nonwoven fabric with very high bulk specific volume, can be manufactured.

Thick fibers generally refer to fibers ≥ 5 dtex, but with the aid of such fibers a material with very high bulk specific volume $\geq 8 \text{ cm}^3/\text{g}$ can be achieved in accordance with the present invention. Bulk is expressed as thickness divided by surface weight of the material (cm^3/g).

According to the present invention there are achieved nonwoven fabrics with a bulk specific volume of 5-15 cm^3/g , especially 8-15 cm^3/g and more particularly 10-15 cm^3/g .

20

One example of a nonwoven fabric manufactured with the aid of the present invention is a nonwoven fabric with a very high bulk of 10-50 cm^3/g , a product, which has very good resilience. 25-50 μm fibers are used in this case. Such fibers, due to their stiffness, are very difficult to entangle by other methods. Such a material is particularly usable as a wicking layer in diapers, but it can be used in a number of different areas, where high bulk and good resilience are desirable properties, such as

25

wipes.

It is of particular advantage to be able to make materials of semi-crystalline polymers, with fibers of thick diameter and/or high modulus of elasticity.

30

The nonwoven fabric produced, by being less dependent on the stiffness of the fiber, can provide a nonwoven fabric consisting substantially 100% of the polymer fiber or the fiber mixture, i.e. a fiber to which no softeners or other additives need to be added as might be required to handle a stiff fiber, for example.

5

The present method for manufacturing nonwoven fabrics is thus less dependent than previous methods on the flexural rigidity of the fiber and, as is evident from the above, provide various possibilities to exploit the potential created. New materials with new properties can be made. For example, a fiber can be optimally stretched before hydroentangling so that it is as stiff as possible, and be entangled. Examples of suitable fibers of this type are polyester fibers and polypropylene fibers. By stretching, the breaking strength of these fibers can be increased to impart new properties to the fiber and the nonwoven fabric produced therefrom and with the aid of the present method, such a fiber can be hydroentangled. A fiber pretreated in this manner is often impossible to hydroentangle by today's methods.

20

The significance of the invention is that fibers having greater stiffness and/or greater thickness than what is normally used in hydroentangling, can be entangled to a high degree of entanglement at reasonable energy levels. Alternatively, fibers of normal stiffness and thickness for hydroentangling can be entangled at lower energy levels and/or to a higher degree of entanglement.

25

The method allows nonwoven fabrics containing very thick fibers to be easily entangled and thus achieve a material of high bulk and good resilience.

An additional advantage is that material can be manufactured at lower cost since the manufacturing cost of synthetic fibers is dimension-related, decreasing with increasing fiber thickness.

An additional advantage is that fibers of very high strength can be entangled into a nonwoven fabric with very good mechanical properties, primarily high wet strength, without the high flexural rigidity of these fibers negatively affecting the degree of entanglement or the energy consumption.

5

In conclusion it can be said that the present invention creates a potential, not only by expanding the number of potential types of fibers as regards polymers and dimensions, but also by making it possible to optimize the fiber components used based on other criteria than limiting the flexural rigidity of the fiber.

This potential can be used for improved material properties (increased bulk, resilience, tensile strength, etc.) or reduced costs in the form of lower energy consumption and/or lower cost for the components used.

As was mentioned previously, the nonwoven fabric can comprise various mixtures of fibers, including mixtures of non-synthetic fibers. The greater the percentage of synthetic polymer fibers included, the greater will be the possibility of using the free potential achieved. The nonwoven fabric will, of course, also have many different properties, depending on the degree of admixture and the type of fiber. On the whole, the present invention provides greater possibilities for optimizing and using new materials.

The nonwoven fabric made according to the present invention can be used as i.a. wiping material for household use or for industrial use, such as major consumers including shops, industries, hospitals, etc. It can also be used for disposable hospital articles, e.g. operation coats, sheets and the like. It can also be used for hygienic purposes, e.g. as components in absorbent products such as sanitary napkins, panty liners, diapers, incontinence products, bed protectors, surgical dressings, compresses and the like. This is particularly true of nonwoven fabrics made according to the present invention with high wet strength. Nonwoven fabrics of high bulk specific

30

volume are particularly advantageous for use as wicking layers in diapers but also as wiping materials for household use.

It is evident from Example 1 below that the tensile strength of the fiber (PLA-fiber) has increased by 20-25% by virtue of the fact that the Tg-temperature was exceeded at hydroentangling. This provides a potential of 20-25%, which can be used in various manners. As can be seen in Example 1, a stronger material can be obtained, but the potential can also be used to provide energy savings and thus savings in costs.

EXAMPLE 1

A foam-formed fiber dispersion consisting of 60% pulp fiber of chemical sulphate pulp and 40% thermoplastic synthetic fiber (1.7 dtex, 19 mm) was formed on a rotating wire. The fiber dispersion was hydroentangled from one side at an energy input of 300 kWh/ton.

The trial encompassed 3 different variants (Trials 1, 2 and 3) of polylactic acid fiber (with $T_g = 50-70^{\circ}\text{C}$) and polyamide 6 (PA, with $T_g = 50^{\circ}\text{C}$; trial 5) as thermoplastic synthetic fiber. At every trial, hydroentangling was performed both with water at room temperature (20°C) and with water heated to 75°C .

As a comparison a trial (trial 4) was made according to the same method with a fiber of polyethylene terephthalat ($T_g = 85^{\circ}\text{C}$).

The tensile strength in dry and wet states (water and tensile solutions) as well as elongation, surface weight, bulk, etc. were measured and the values are displayed in Table 1 below.

T_g was measured with the aid of a Perkin Elmer DSC 7 and the measurement was done from room temperature to 50°C over the melting point.

The values of initial modulus were obtained in the following manner. The tensile test was performed on a Lenzing Vibrodyn with a pulling speed of 50 mm/min. and a gauge length of 10 mm. A weight of 100 mg was used to pretension the fiber. The initial modulus was calculated manually by drawing in the tangent to the tensile testing curves in the linear range. The values of the initial modulus given in the table are at room temperature.

In the dry state, the fibers have a relatively high friction against each other and the dry strength of the nonwoven fabric largely depends on the mechanical properties of the individual fibers, such as tenacity, elongation and initial modulus.

As can be seen in Table 1, the strength in the dry state is hardly affected at all or to a lesser extent, which indicates that the fibers have reassumed their original mechanical properties after the momentary heat treatment to which they were subjected.

When tensile tests are performed in water, the fibers will slide more easily against each other and the degree of mechanical bonding (entangling) will assume greater importance for the mechanical properties of the nonwoven fabric. Table 1 shows that for all of the trials the tensile index was somewhat greater for those materials which have been hydroentangled with hot water.

For tensile testing in the tenside solution, the friction between the fibers was essentially removed, and this made the degree of entanglement the dominant factor for the mechanical properties of the nonwoven fabric. As is evident from Table 1, there is a significant increase of between 20 and 25% for PLA and more than 50 % for PA in the tensile index when the material was hydroentangled in hot water.

The values of the stiffness index were essentially unchanged (certain of them have increased somewhat, certain have decreased somewhat) and this shows that the type of bonding is unchanged. If the increase in the tenside strength were dependent on

the fact that the fibers had been thermally bonded to each other during the heat treatment, this would also be revealed as a dramatic increase in the rigidity index. In order to achieve thermobonding of polylactic acid, significantly higher temperatures are required however.

5

What has been achieved is thus a nonwoven fabric where the fibers have their original mechanical values, but where the structure has been changed in such a way that the fibers have been entangled to a higher degree.

Table 1

Trial No.	Poly-mer fiber	Temperature °C	Surface weight g/m ²	Thickness µm	Bulk specific volume cm ³ /g	Stiffness Index Nm/g	Tensile Index Dry Nm/g	Stretch Index %	Work to rupture index J/g	Tensile Index Water Nm/g	Tensile Index Tenside Nm/g	Initial modulus
1	PLA	20	87.7	474	5.4	114	14	74	5.8	12.1	10	210
	PLA	75	91.8	408	4.4	100	14	57	6	12.3	12.1	210
2	PLA	20	91	521	5.7	73	14	44	4.3	8.8	7.4	
	PLA	75	89.8	493	5.5	65	14	47	4.5	9.3	9.1	
3	PLA	20	85.6	490	5.7	71.1	19.9	52	7.2	13.9	11.1	502
	PLA	75	91.3	487	5.3	89.7	19.2	56	7.3	15.4	13.4	502
4	PET	20	85.3	499	5.8	51.6	23.5	66	8.7	20.3	11.8	
	PET	75	85.6	476	5.6	55	23.5	62	8.5	20.8	13.2	
5	PA	20	90.8	503	5.5	78.4	25.6	81.3	11.9	14.1	5.6	
5	PA	75	88.1	466	5.3	123.4	30.0	75.6	13.1	18.1	8.9	

CLAIMS

1. Method of hydroentangling polymer fibers to manufacture a nonwoven fabric,
characterized in that the polymer fiber, at the moment of hydroentangling, is
5 imparted a temperature equal to or exceeding the glass transition temperature
(Tg) of the polymer fiber and being less than the melting point of the polymer
fiber.
2. Method according to claim 1, **characterized in** that the polymer fiber has an
10 initial modulus ≥ 50 cN/tex, at room temperature.
3. Method according to claim 1, **characterized in** that the polymer fiber has an
initial modulus ≥ 100 cN/tex, at room temperature.
- 15 4. Method according to claim 3, **characterized in** that the polymer fiber has an
initial modulus of 100 – 2000 cN/tex, especially 500 – 1500 cN/tex, more
particularly 200 – 750 cN/tex, and even more particularly 250 – 600 cN/tex, at
room temperature.
- 20 5. Method according to any of claims 1 – 4, **characterized in** that the temperature
is achieved with the aid of hot or superheated water.
6. Method according to any of claims 1 – 4, **characterized in** that the temperature
is achieved with the aid of IR-heat.
- 25 7. Method according to any of claims 1 – 4, **characterized in** that the temperature
is achieved with the aid of microwaves.
8. Method according to any of claims 1 – 7, **characterized in** that the polymer
30 fiber has a glass transition temperature (Tg) of $\geq 20^{\circ}\text{C}$.

9. Method according to any of claims 1 – 8, **characterized in** that the polymer fiber has a glass transition temperature (T_g) of 20 - 100°C, especially 50 - 70°C.
- 5 10. Method according to any of claims 1 – 9, **characterized in** that the polymer included in the polymer fibers comprises polyester, polylactic acid, polyamide or polypropylene, or copolymers or mixtures thereof.
- 10 11. Hydroentangled nonwoven fabric obtainable by the method of any of claims 1-10 comprising polymer fibers, **characterized in** that the polymer fibers in the nonwoven fabric have a glass transition temperature (T_g) of 20 - 100°C and an initial modulus of 200 – 750 cN/tex at room temperature.
- 15 12. Nonwoven fabric according to Claim 11, **characterized in** that the polymer fibers in the nonwoven fabric have an initial modulus of 250 – 600 cN/tex at room temperature.
- 20 13. Nonwoven fabric according to any of claims 11-12, **characterized in** that the polymer fibers in the nonwoven fabric have a glass transition temperature (T_g) of 50 - 70°C.
14. Nonwoven fabric according to any of claims 11 – 13, **characterized in** that the nonwoven fabric has a bulk specific volume of $\geq 8 \text{ cm}^3/\text{g}$.
- 25 15. Nonwoven fabric according to claim 14, **characterized in** that the nonwoven fabric has a bulk specific volume of $8 - 15 \text{ cm}^3/\text{g}$, especially $10 - 15 \text{ cm}^3/\text{g}$.
- 30 16. Nonwoven fabric according to any of claims 11– 15, **characterized in** that the polymer included in the polymer fibers comprises polyester, polylactic acid, poly-amide or polypropylene, or copolymers or mixtures thereof.

17. Nonwoven fabric according to one of Claims 10 – 15, **characterized in that** the polymer included in the polymer fibers comprises polyester, polylactic acid, poly-amide or polypropylene, or copolymers or mixtures thereof.

ABSTRACT

The invention relates to a method of bonding polymer fibers into a nonwoven fabric and a nonwoven fabric manufactured with the aid thereof. According to the invention, the polymer fibers are subjected to hydroentangling, the polymer fibers at the moment of hydroentangling being imparted a temperature exceeding the glass transition temperature of the polymer fiber, but being less than its melting point.

5

000510-011

Attorney's Docket No.

**COMBINED DECLARATION AND POWER OF ATTORNEY
FOR UTILITY OR DESIGN PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

FIBER BONDING

the specification of which (check only one item below):

- ☐ is attached hereto.
- ☐ was filed as United States application
Number _____ on _____
and was amended _____ on _____ (if applicable).
- ☒ was filed as PCT international application
Number PCT/SE00/01670 on 31 August 2000
and was amended _____ on 17 October 2001 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §§119 (a)-(d), 172 or 365 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §§119(a)-(d), 172 or 365:					
COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §§119, 172 or 365		
Sweden	9903075-1	01 September 1999	X	Yes	No
				Yes	No
				Yes	No
				Yes	No
				Yes	No

Combined Declaration and Power of Attorney
for Utility or Design Patent Application
Attorney's Docket No. 000510-011
Page 2 of 2

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

William L. Mathis	17,337	Eric H. Weisblatt	30,505	Bruce T. Wieder	33,815
Robert S. Swecker	19,885	James W. Peterson	26,057	Todd R. Walters	34,040
Platon N. Mandros	22,124	Teresa Stanek Rea	30,427	Ronni S. Jillions	31,979
Benton S. Duffett, Jr.	22,030	Robert E. Krebs	25,885	Harold R. Brown III	36,341
Norman H. Stepno	22,716	William C. Rowland	30,888	Allen R. Baum	36,086
Ronald L. Grudziecki	24,970	T. Gene Dillahunty	25,423	Brian P. O'Shaughnessy	32,747
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Kenneth B. Leffler	36,075
Alan E. Kopecki	25,813	B. Jefferson Boggs, Jr.	32,344	Fred W. Hathaway	32,236
Regis E. Stuter	26,999	William H. Benz	25,952	Wendi L. Weinstein	34,456
Sarduel C. Miller, III	27,360	Peter K. Skiff	31,917	Mary Ann Dillahunty	34,576
Robert G. Mukai	28,531	Richard J. McGrath	29,195	Donna M. Meuth	36,607
George A. Hovanec, Jr.	28,223	Matthew L. Schneider	32,814	Mark R. Kresloff	42,766
James A. LaBarre	28,632	Michael G. Savage	32,596		
E. Joseph Gess	28,510	Gerald F. Swiss	30,113		
R. Darny Huntington	27,903	Charles F. Wieland III	33,096		



21839

and:

Address all correspondence to:



21839

Ronald L. Grudziecki
BURNS, DOANE, SWECKER & MATHIS, L.L.P.
P.O. Box 1404
Alexandria, Virginia 22313-1404

Address all telephone calls to: William C. Rowland at (703) 836-6620.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST INVENTOR	Lars Fingal
Signature	<i>Lars Fingal</i>
Date	20 February 2002
Residence (City, State, Country)	Göteborg, Sweden <i>SEK</i>
Citizenship	Swedish
Mailing Address	Uddevallplatsen 12
City, State, ZIP, Country	S-416 70 Göteborg, Sweden
FULL NAME SECOND INVENTOR, IF ANY	Anders Strålin
Signature	<i>Anders Strålin</i>
Date	20 February 2002
Residence (City, State, Country)	Torslanda, Sweden <i>SEK</i>
Citizenship	Swedish
Mailing Address	Andalen 95
City, State, ZIP, Country	S-423 38 Torslanda, Sweden